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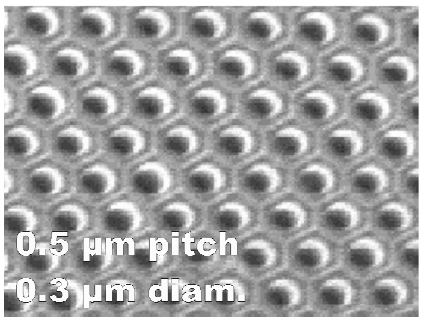
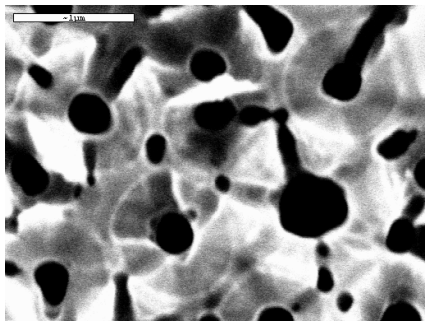
LAPD MCP Godparent Review
Argonne National Laboratory
October 4, 2010

AAO MCP SUBSTRATES DEVELOPMENT AT SYNKERA

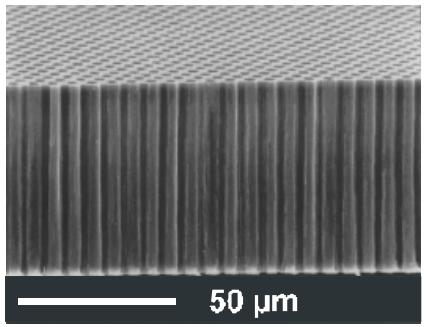
CUMULATIVE STATUS UPDATE
Oct. 10, 2009 - Oct 4, 2010

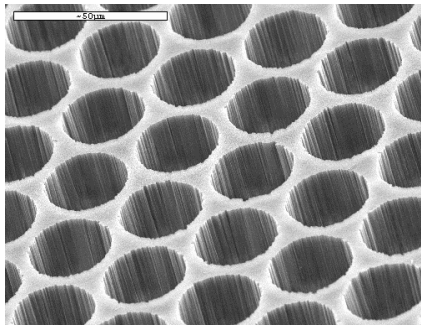
AAO AS MCP SUBSTRATE



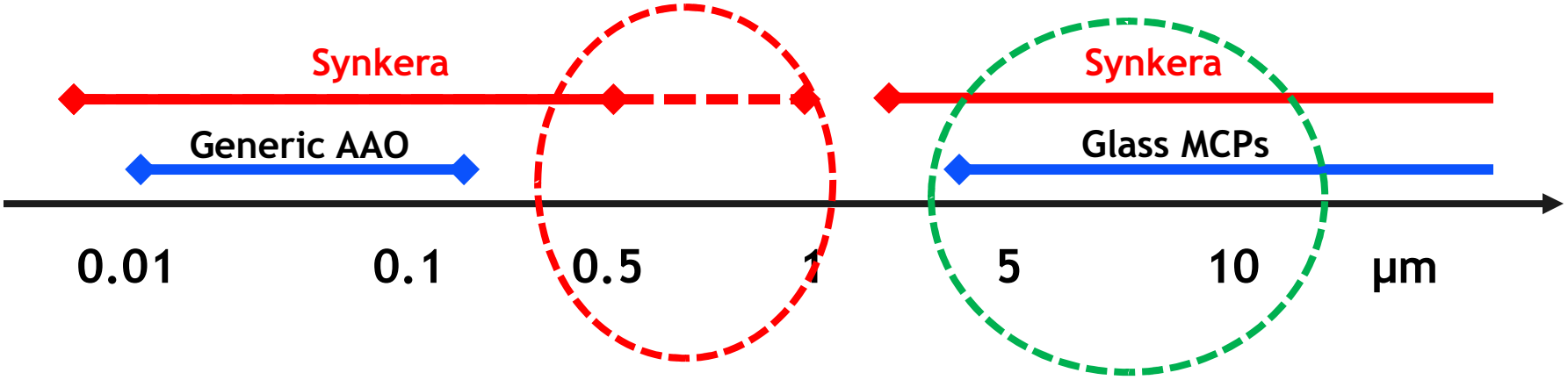


intrinsic pores





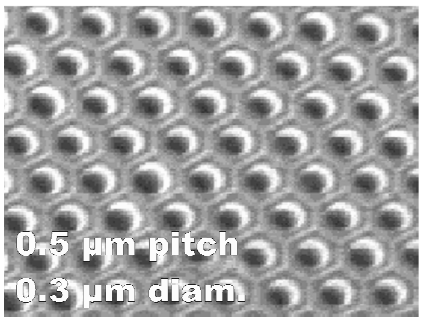
micromachined channels

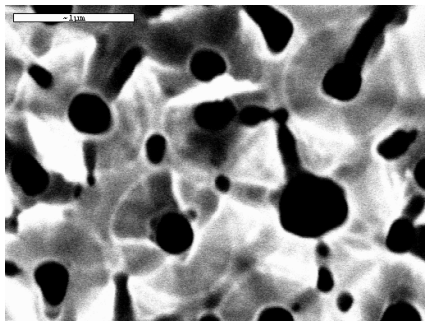


LAPD and NSF Phase II

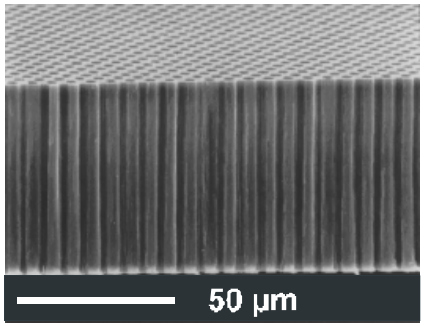
RELATED CURRENT EFFORT

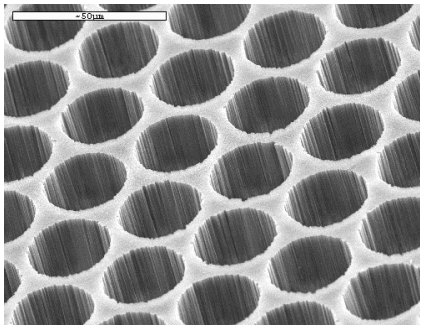
AAO AS MCP SUBSTRATE



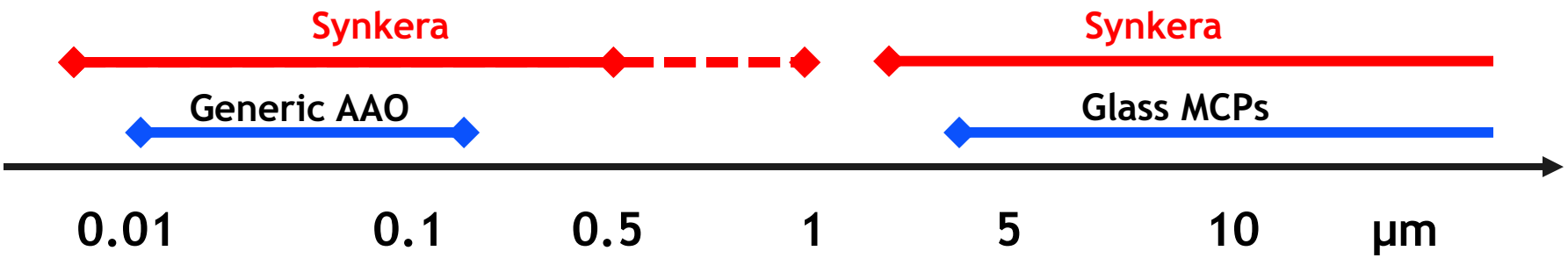


intrinsic pores





micromachined channels



Main challenges:

- Achieving channel diameter in the 0.5 – 2 µm range:
  - Intrinsic - stable anodization at 400-600 V
- Scaling to dimensions and formats of detector targeted by LAPPD
  - Current effort
- Scaling to manufacturing of 8”x8” substrates at targeted cost

SYNKERA LAPD SUBCONTRACT

Overall goal: develop ceramic MCPs substrates for low-cost large area detectors.

The project benefits from prior and related IP, established facilities and on-going R&D and scale-up efforts at Synkera.

Year 1 – Development of Required Channel Structure

- Objective: maximize channel diameter and enable a funnel-shaped opening, while maintaining well-aligned channels.
- Deliverables:
  - Demonstrate AAO with channel diameter ≥0.5 μm, OAR ≥60%, L/D of 50-100
  - MCP substrates for the LAPD project team (32.8 mm, qty ≥ 15) targeting above specs
  - Initial cost projections for 8”x8” AAO substrates

Year 2 Option – Support of MCP Development and Scale-Up

- Objective: enable targeted MCP performance via development of AAO substrates; limited scale-up to validate the size (8”x8”) and cost reduction potential.
- Deliverables:
  - Demonstrate channel diameter ≥0.7 μm, funnel-shaped opening, OAR≥65%, and L/D 40-100.
  - MCP substrates for the LAPD project team (32.8 mm, qty ≥ 40) targeting above specs
  - Optional: scaled 8”x8” “demo” substrates
  - Validated cost projections for 8”x8” AAO substrates.

Year 1 Summary

Task 1: Develop targeted AAO structure - **COMPLETED**

- New processes for high-voltage anodization (up to 500V) established
  - *Reduced voltage ramp (function of substrate size)*
  - *Improved (not perfect yet) pore uniformity and alignment*
- Targeted AAO parameters demonstrated in small samples
  - *pore period >1μm and diameter ~0.5 μm*
  - *thickness of 0.02 - 0.1 mm and L/D = 40 - 100*

Task 2: Deliver 33 mm prototypes - **IN PROGRESS**

- Equipment, tooling & processes scaled up to 33 mm
- Processing scale-up and validation for 33 mm in progress
- Prototypes delivery
  - *Batch 1: 4 substrates Ø32.8 mm - ANL, 2 substrates Ø25 mm – Arradiance*
  - *Batch 2: 3 substrates Ø32.8 mm - ANL; 4 substrates Ø32.8 mm - Arradiance*
  - *Batch 3: 6-10 substrates in processing; to be delivered by the end of Year 1*
  - *Optional: demo 8" x 8" substrate*

Task 3: Initial cost analysis - **COMPLETED**

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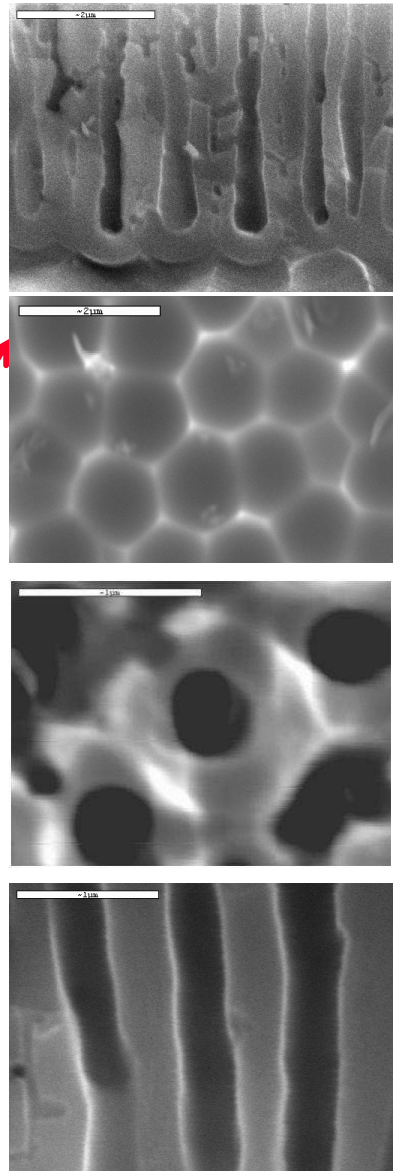
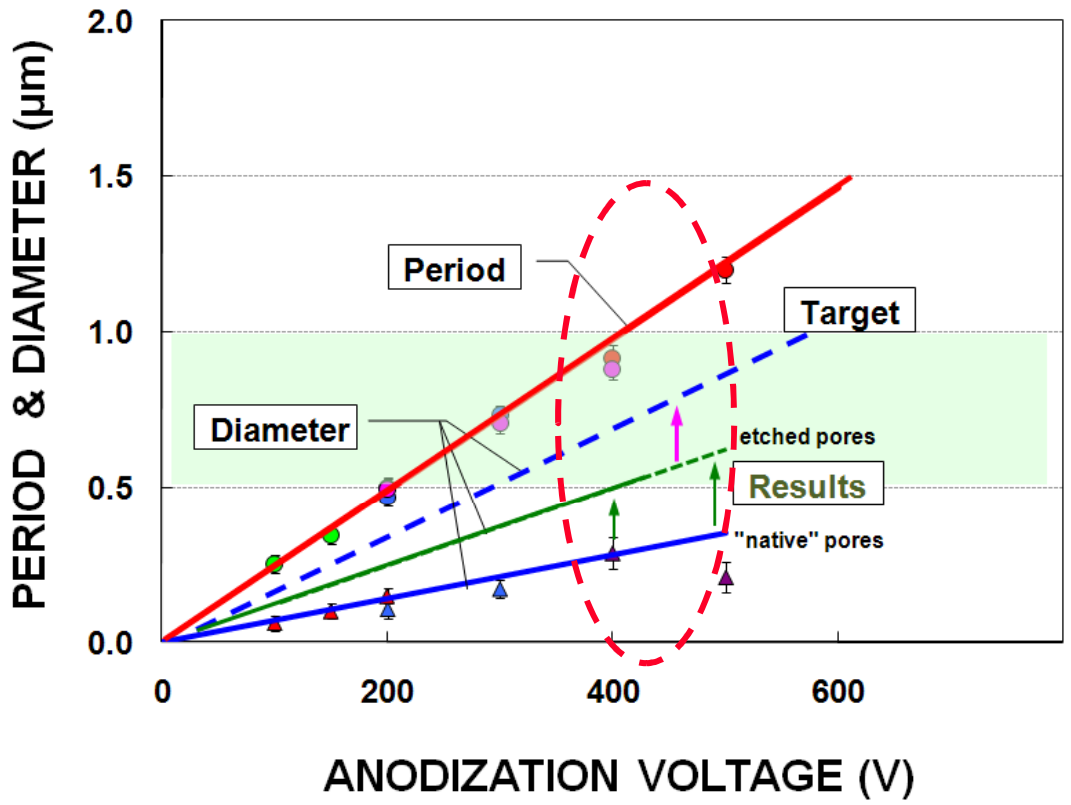
Task 1: DEMONSTRATING AAO STRUCTURE

TARGET	PREREQUISITE	DEMONSTRATED	GOAL
channel period >1μm	anodization at 400 - 600 v	• 0.8-1 μm @ 400V, reliable process; • 1.2 μm @ 500V	eliminate voltage ramp for uniform period across aao
channel diameter ≥0.5 μm	period >1 μM, conformal etch	~0.5 μm @ 400V	achieve 0.7 μm for 500V; improve uniformity at 400V
L/D 50-100	appropriate thickness (<20 μm AAO is too fragile)	for 50-100μm thick AAO, L/D = 100-200	improve growth rate uniformity
OAR ≥ 60%	conformal pore etching	55-60% for 400V, reliable process	≥ 60% depending on mechanical prop.
funnel entrance	-	default feature	at least 45° angle



ACHIEVING CHANNEL DIAMETER ≥ 0.5 µm



Pore Period:

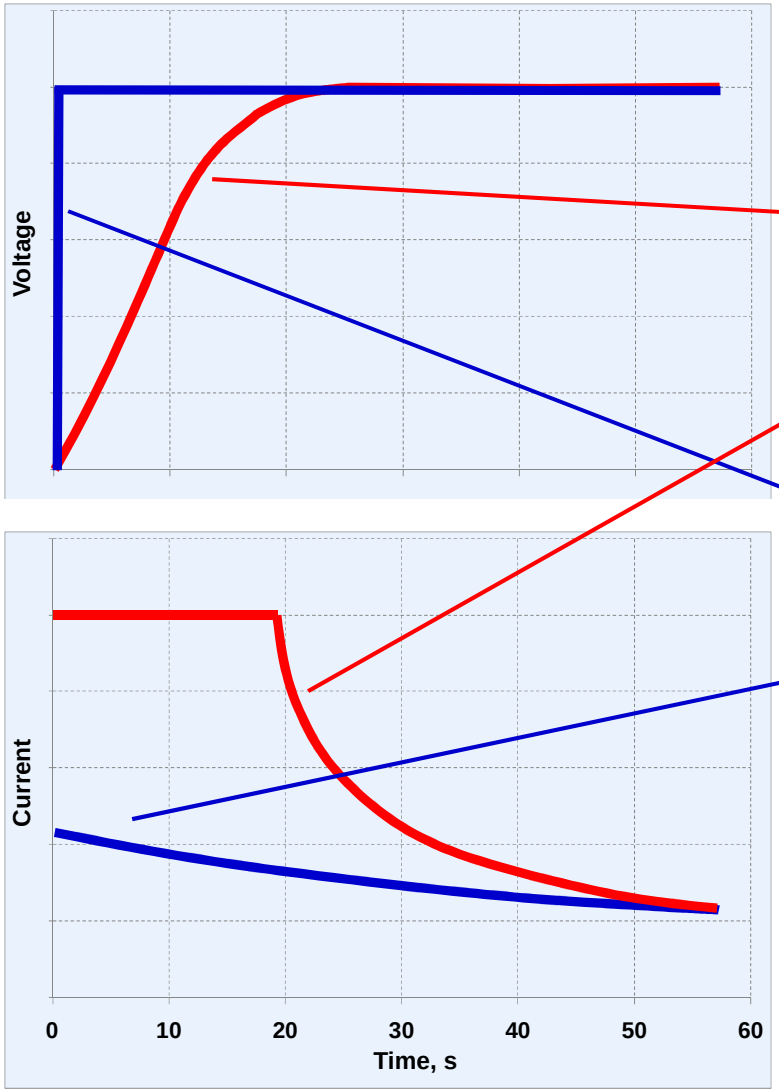
- Proportional to anodization voltage
- Does not depend on electrolyte, temperature

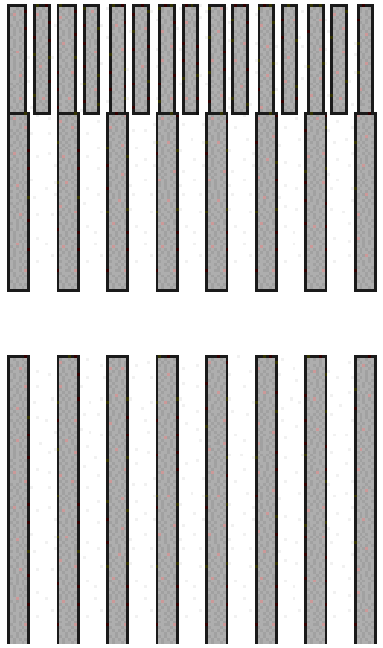
Pore Diameter:

- Increases with voltage, [H+] and temperature

Starting point - patent-pending processes developed in 2006 - 2009 under NSF SBIR

Voltage ramp → non-uniformity of channel period



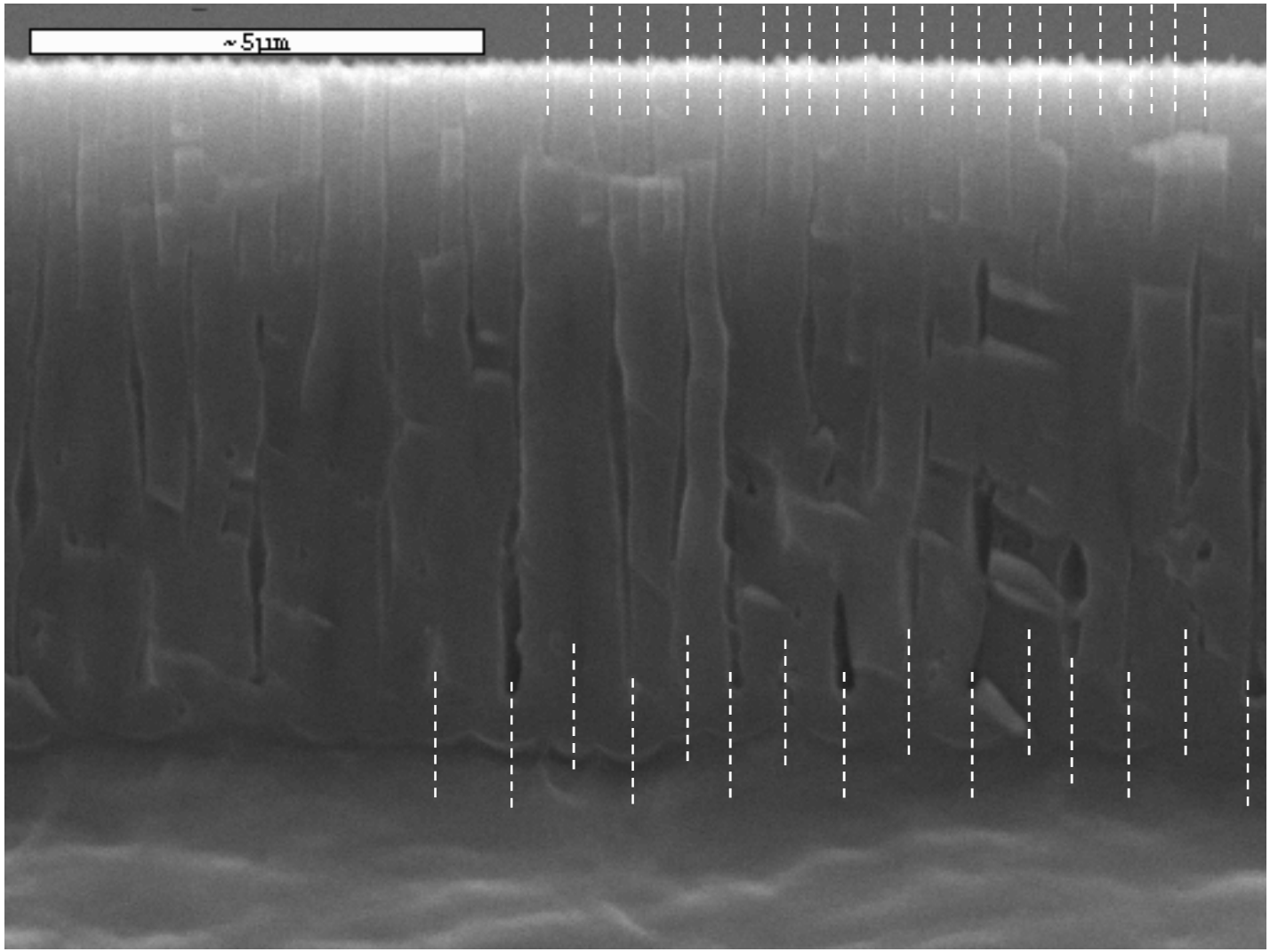


Current

Targeted

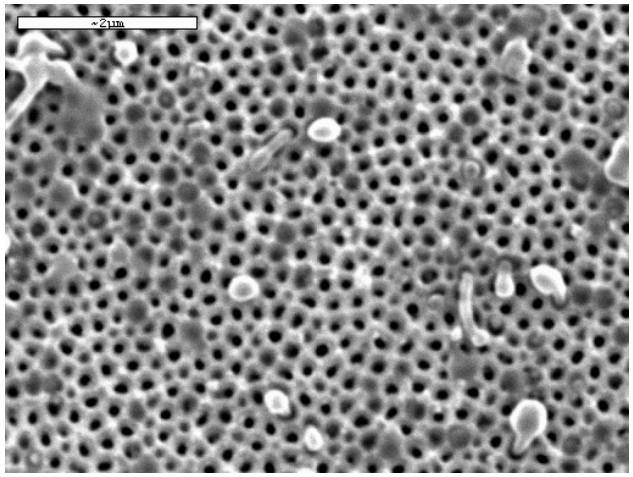
- AAO pore period ~ Voltage (U)
- U ramp leads to surface layer with smaller pore period & diameter
- This is the main challenge in forming desired structure

Effect of Voltage Ramp

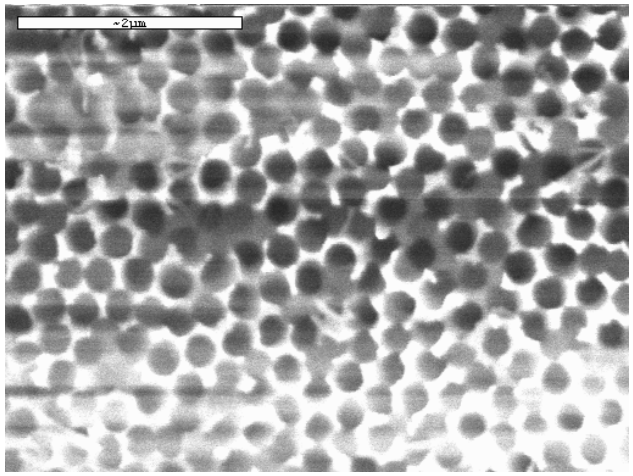


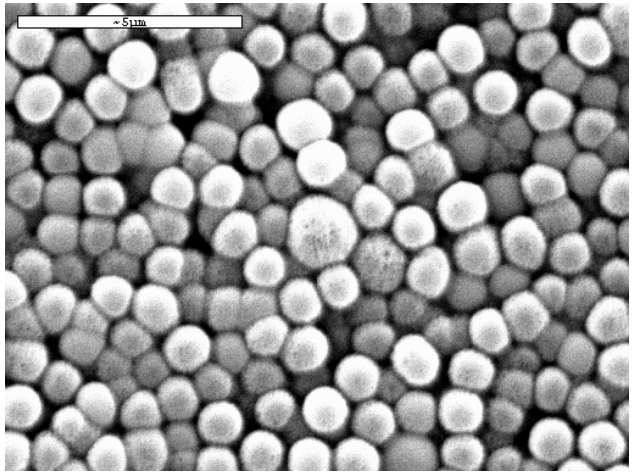
Before Etch

After Etch

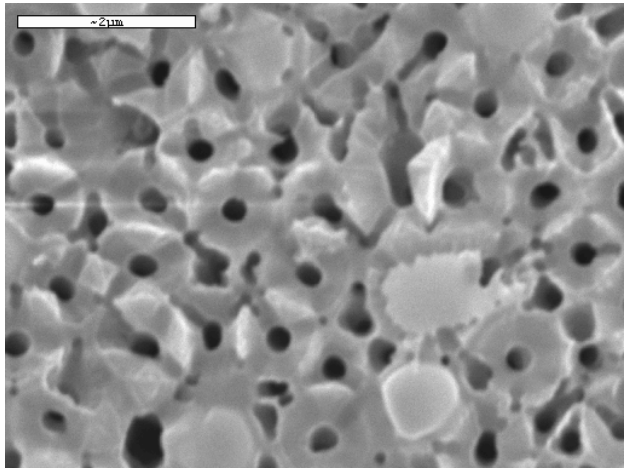


"Solution" Surface





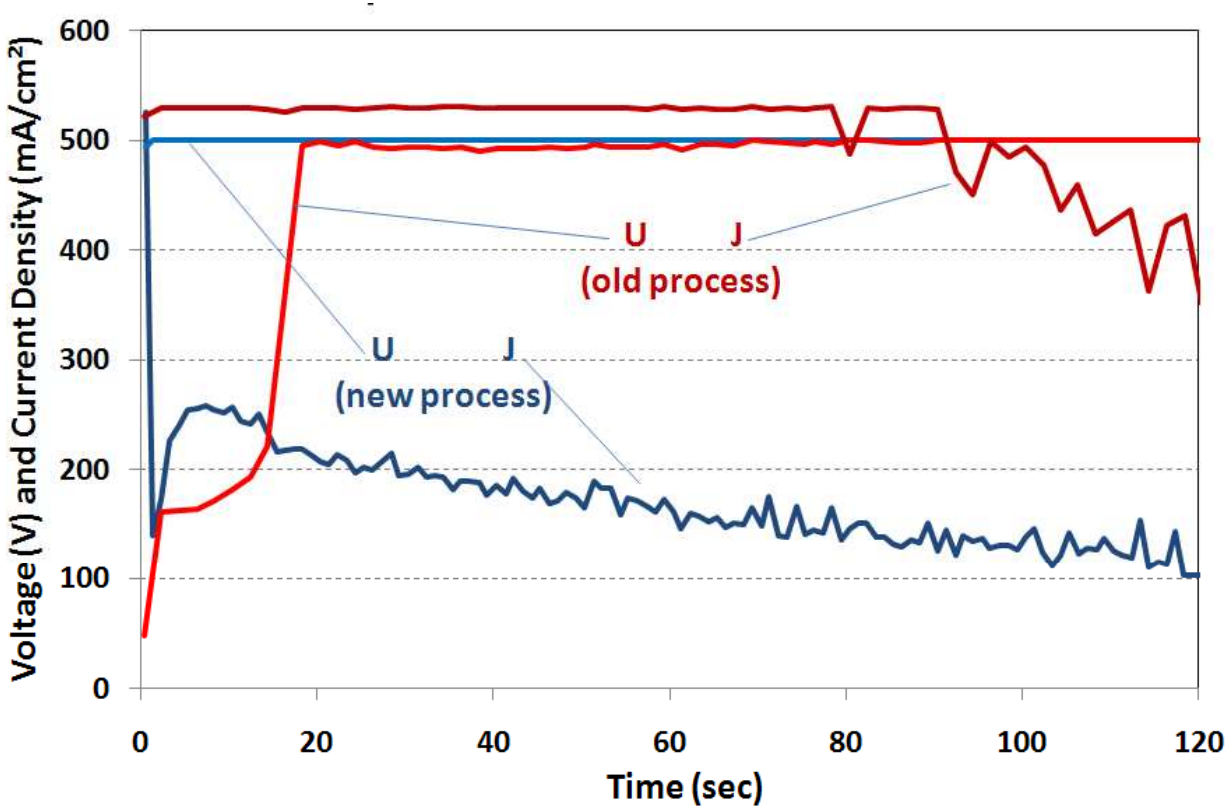
"Barrier Layer" Surface



BL closed; structure is impermeable to gases

BL open; structure is permeable to gas flow

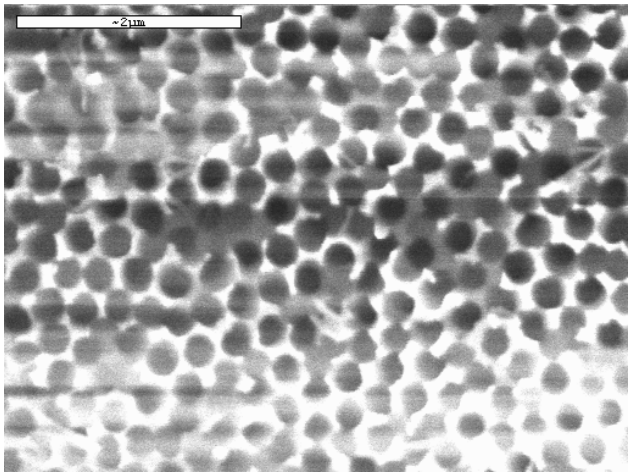
HIGH VOLTAGE ANODIZATION



- For the first time, no voltage ramp even at 500V (small thin samples)
- Initial instability and arching during process start
- Reduced current oscillations – better structure
- Very rapid drop in anodization current -> very slow growth rate

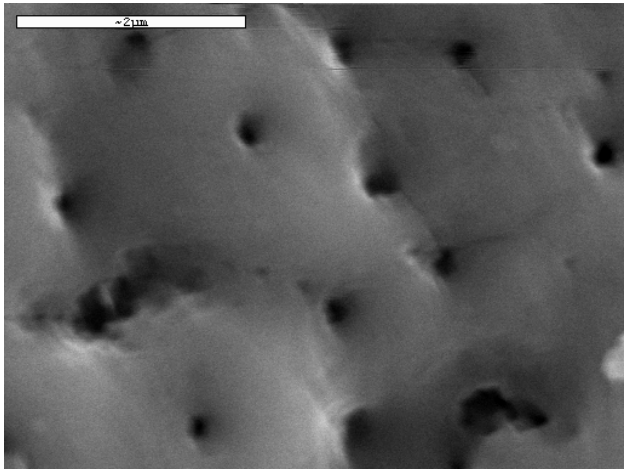
AAO Surfaces With and Without Voltage Ramp

With Voltage Ramp

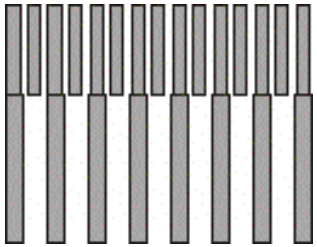


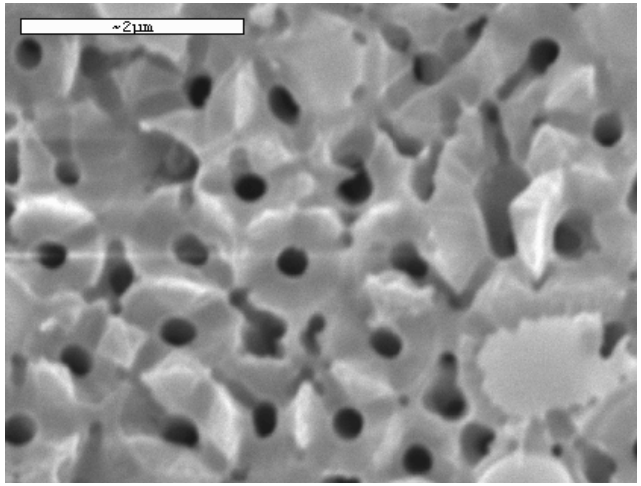
Solution Side

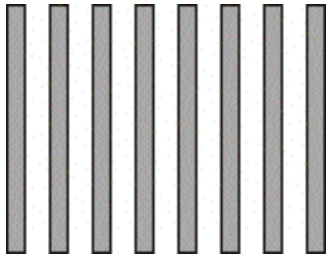
No Voltage Ramp



Barrier Layer Side

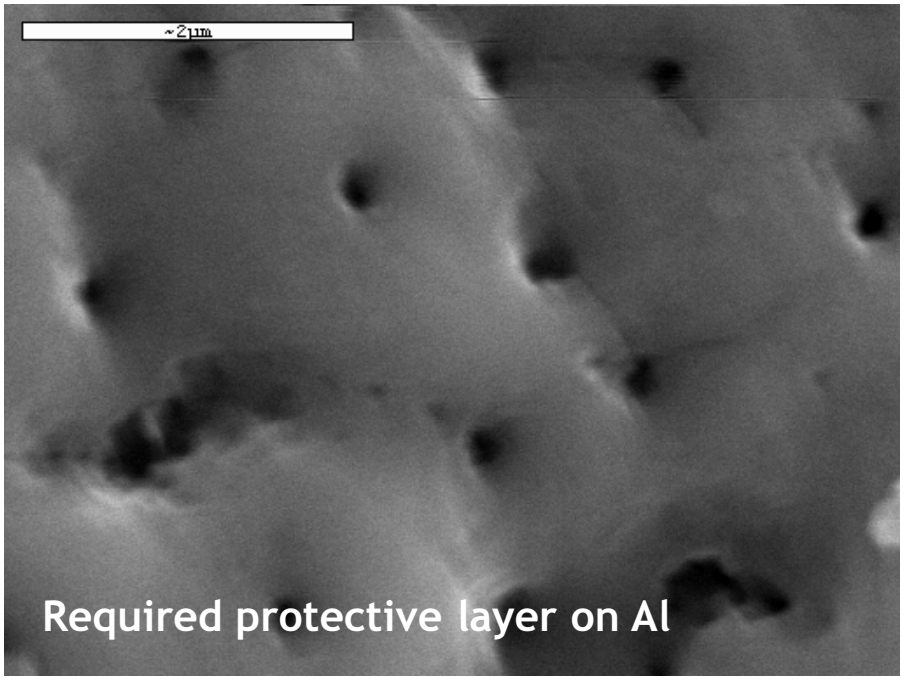




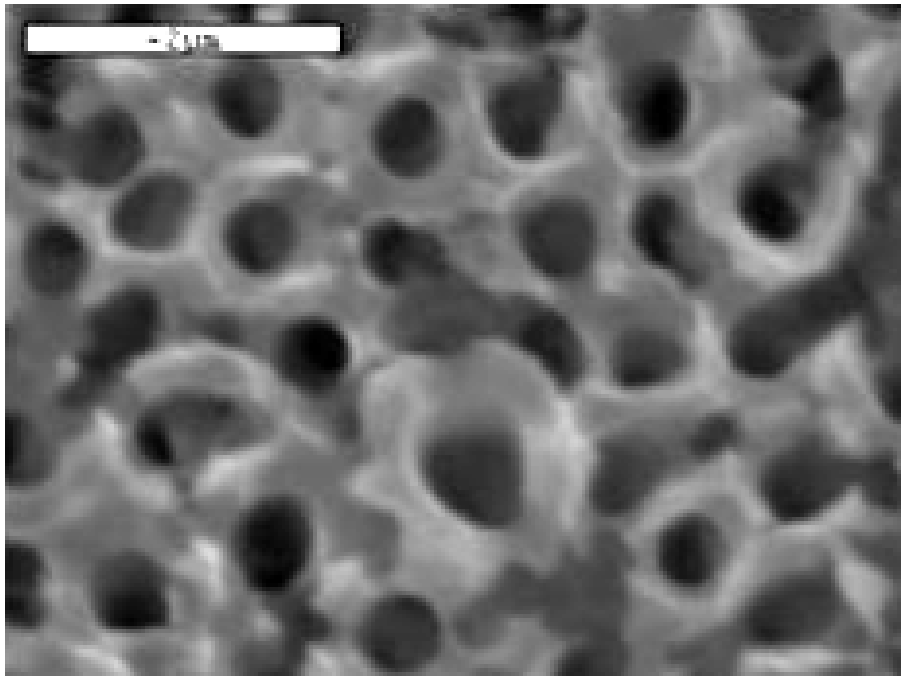


Best AAO Structure Achieved to Date

Solution Side



Barrier Layer Side



- Pore period 1.0-1.2 μm (reuires protective layer on solution side)
- Pore diameter 0.2-0.3 μm; 0.4-0.5 μm after conformal etching
- Funnel-like channel opening
- Good pore structure in select conditions
  - Thickness limitation for best structure

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TASK 2: FABRICATION OF PROTOTYPE SUBSTRATES

Status

- Dedicated anodization setup built, up to 600V
- 400V process selected for initial scaling to 32.8 mm
- 11 substrates Ø32.8 mm total delivered
- Additional substrates in processing

Challenges in scaling high voltage processing

- Voltage ramp problem worsens with larger size due to high current
- Achieving required thickness while maintaining aligned and uniform channels
- Increasing the pore diameter leads to greater fragility

Approaches to mitigate

- Using novel electrolytes & anodization regimes
- Adequately masking and protecting Al surface to avoid breakdown & arching
- Use different chemistries and/or conditions at different AN stages

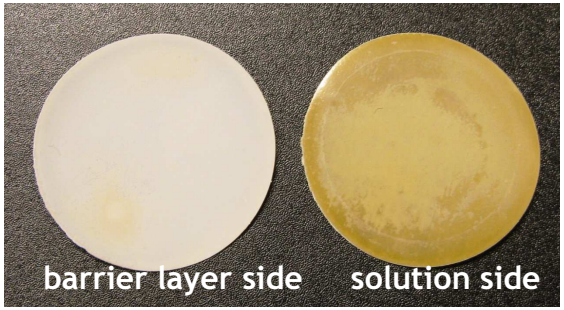
SCHEDULE OF DELIVERABLES

Deliverable	Completion Date	Status	Payment
1. Report on setting up equipment & tooling for fabricating 32.8 mm AAO substrates; initiating MCP development.	11/30/2009	COMPLETED	$30,000
2. Progress report and the 1st batch of the 32.8 mm AAO substrates to the project team.	~~01/31/2010~~ 3/24/2010	COMPLETED	$30,000
3. Progress report that demonstrates AAO with channel diameter ≥0.5 µm.	~~03/31/2010~~ 3/24/2010	COMPLETED	$30,000
4. Progress report and the 2nd batch of the 32.8 mm AAO substrates to the project team.	~~05/31/2010~~ 05/~~10~~20/2010	COMPLETED	$30,000
5. Progress report that demonstrates AAO with OAR≥60%, funnel-shaped entrance and channel diam. ≥0.5 µm. Summary presentation for DOE review	07/31/2010 06/10/2010	COMPLETED	$30,000
6. Progress report including cost projections for 8"x8" AAO substrates Remaining 32.8 mm AAO substrates to the project team. Acceptance by ANL of all work covered by the contract. Optional: demo 8" x 8" AAO substrate for mechanical evaluation.	10/31/2010 ~~9/1/110~~ 10/31/2010 ?	IN PROGRESS	$25,000
Total for Year 1			*$175,000*

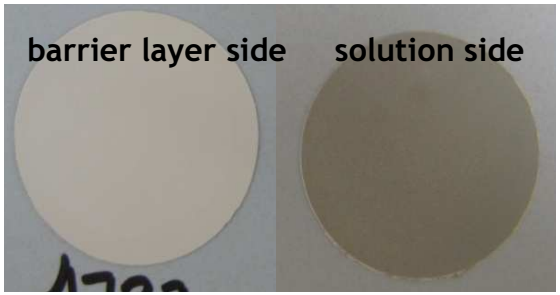
Delivered To Date

Overall size:	32.8 mm
Active area:	24.5 mm
Thickness :	100 (#1-2) µm 50 (#3)
Pore Period:	1 – 1.2 µm (400V)
Pore Diam.:	0.5 µm targeted
OAR:	TBD
Annealing:	500°C, 750°C

Batch 1



Batch 2



Batch 1: DELIVERED (x8)

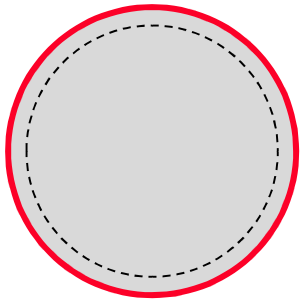
- voltage ramp; surface has smaller channels
- opposite sides are different in morphology
- some discoloration from processing residue

Batch 2: DELIVERED (x 7)

- improved processing
- voltage ramp eliminated
- more uniform surface than in Batch 1

Batch 3: IN PROGRESS

- larger pore diameter and improved surface structure
- Reduced aspect ratio for Arradiance (thickness 50 µm and 100 µm)



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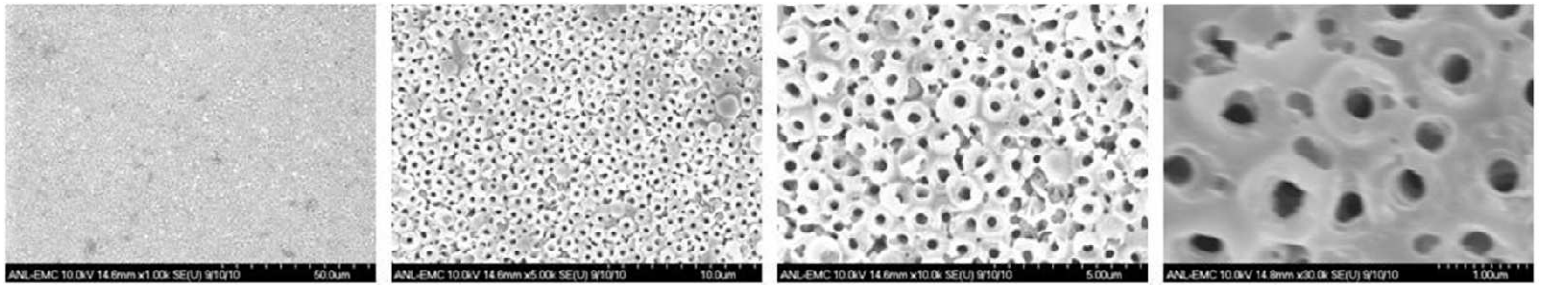
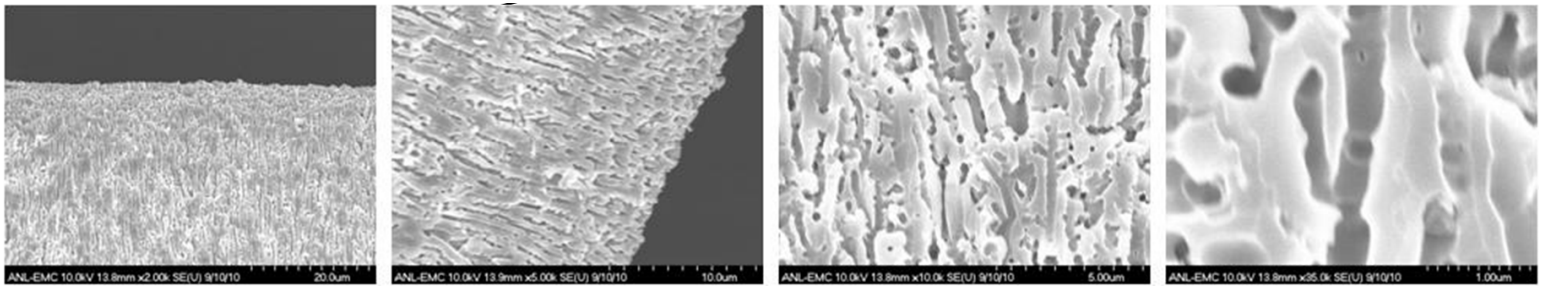
ANL SEM Images of Batch 2 Prototypes

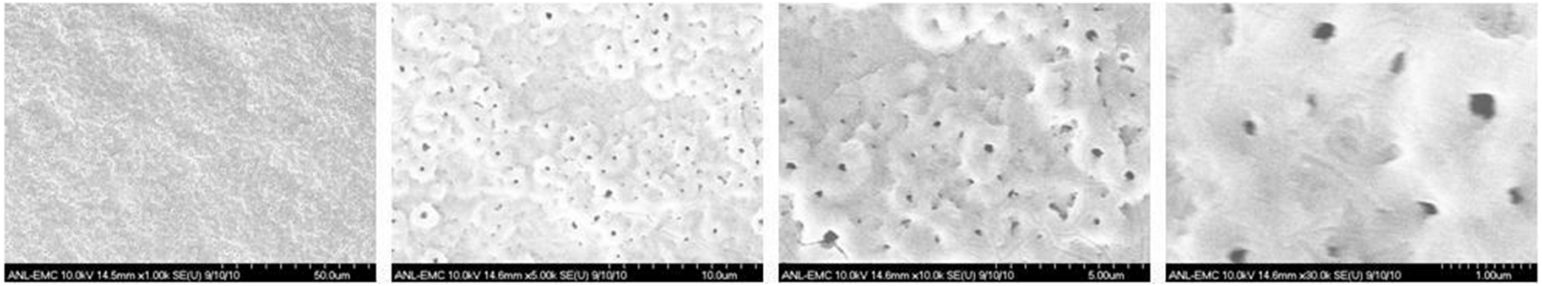
barrier layer side



cross-section



solution side



- pore diameter 0.2-0.3 μm (barrier layer)
- pore diameter 0.1 μm (solution side)
- protective layer visible

COMMENTS ON THE STRUCTURE AND TESTING

Aspect ratio

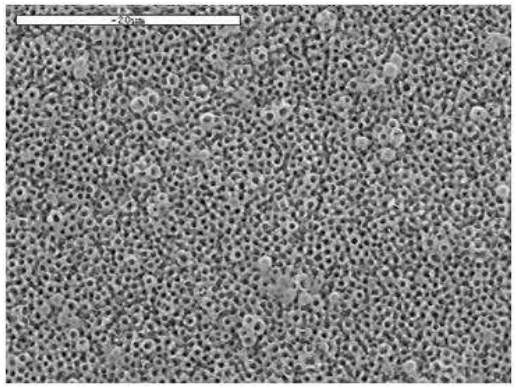
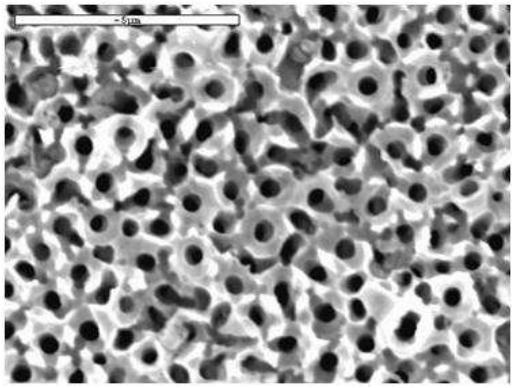
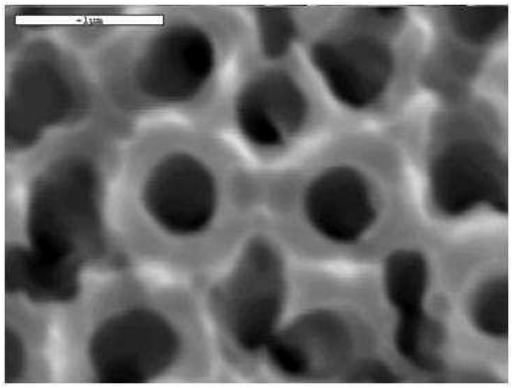
- Current thickness is 100 μm, target channel diameter 0.5 μm
- Resulting AR of 200 could be too high for testing at Arradiance or ANL
  - Neal suggested to made 50 μm thick AAO for target AR of 100 - will be included into the 3rd batch
  - Samples with AR of 200 could be tested at Berkeley at higher voltages
- Concern (Jeff): 50 μm AAO may be bent during ALD based on previous work
  - We anneal AAO to 900°C, which could alleviate bending even with 50 μm AAO
  - There has not been any bending with Synkera 100μm thick substrates so far

Channel Entrance & Structure

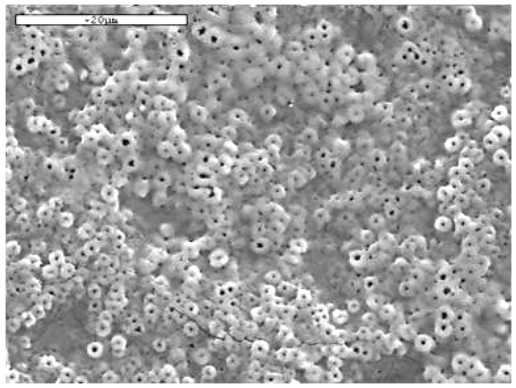
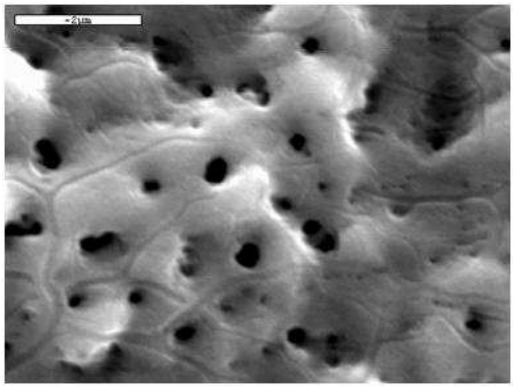
- The surface topology of the two sides of AAO made at high voltage is different
- This may have lead to partial blockage of the channels on one side in batch #1 after ALD and electroding
  - Suggestion: AAO-based MCP should be tested in two orientations, with both sides facing the front.
  - We are working on further alleviate this issue in batch #3
- The overall structure of the pores is not ideal yet; we continue to work on the process development with the aim to improve the structure

Additional Etching of Batch 2

barrier layer side

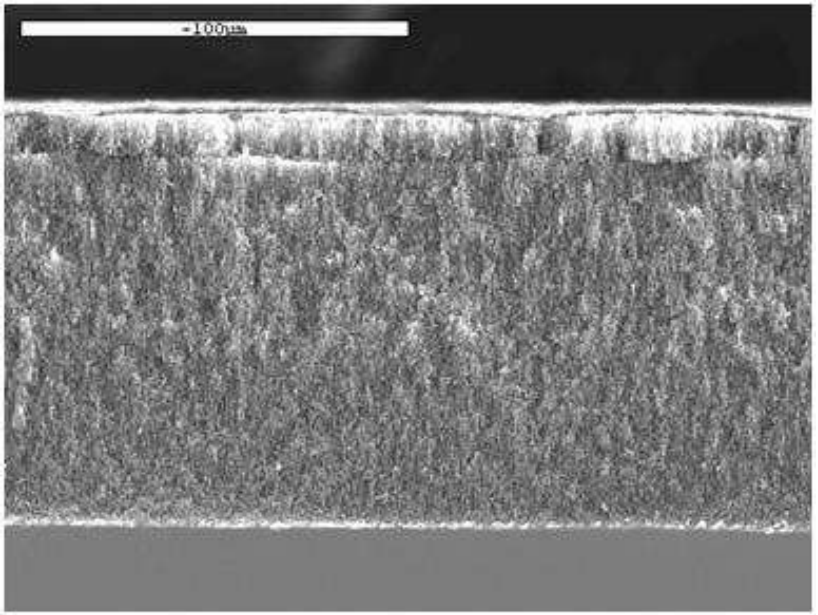




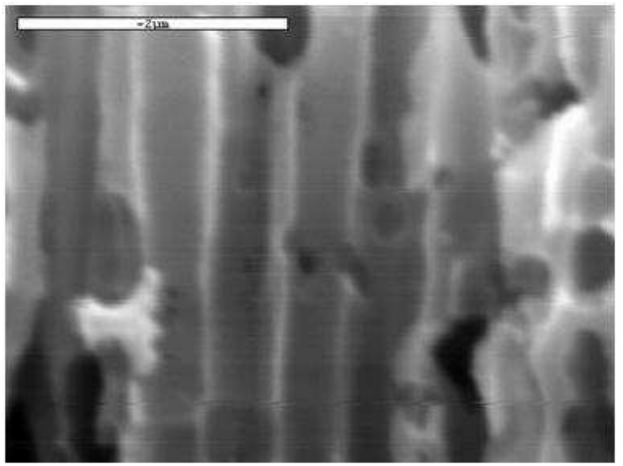
solution side

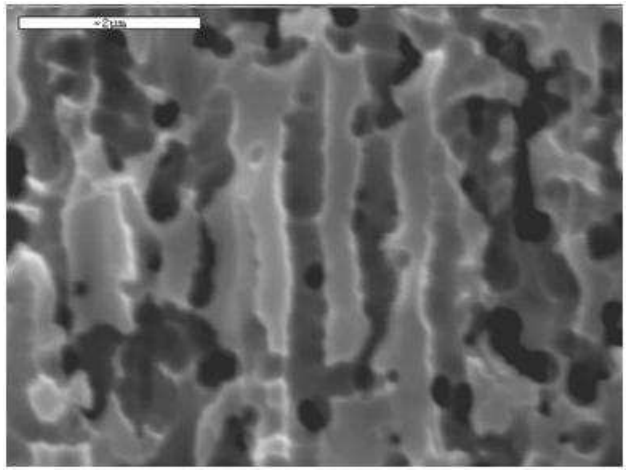



- pore diameter ~0.4-0.5 µm (barrier layer)
- pore diameter 0.2-0.4 µm (solution side)

Structure Evolves with Thickness







- Protective layer on the top (solution side)
- Initial layer grown in different electrolyte separates with etching
- Q: should these be polished off?
- Pore morphology changes with decreasing growth rate as pores get deeper

SUMMARY OF THE DELIVERABLES

Batch / Delivery	Batch 1, March '10	Batch 2, June'10	Batch 3, 10/31/10
Structure	Non-ideal structure. Smaller channel entrance on one side due to voltage ramp	Voltage ramp eliminated. Differences in topology exists between the 2 faces; Insufficient etch, tortuous pore structure	Incl. 50 μm for AR=100. Target better structure and larger channel diameter
ALD of Resistive Coating	ANL: >GOhm Arradiance: ~100 MOhm with 100-200 nm resistive film	ANL: 100 MOhm with ANL1 (x2) Arradiance: TBD	-
Electroding	ANL: done (mat-l, thickness?) Arradiance: done (mat-l, thickness?)	ANL: done for 2 substrates (mat-l, thickness?) Arradiance: TBD	-
Testing	ANL: no testing done Arradiance: no gain - AR 200 too high - possible channel blockage	**UC Berkeley: no gain** **Arradiance: AR too high**	-
Comments	Batch 1 targeted development of resistive ALD coatings and was not optimized for functional MCP	Appears better that 1st batch. Use both sides as a front surface during testing. Use test results for development of batch 3.	**Main target - demonstrate gain in AAO MCPs**

TASK 3: COST PROJECTIONS

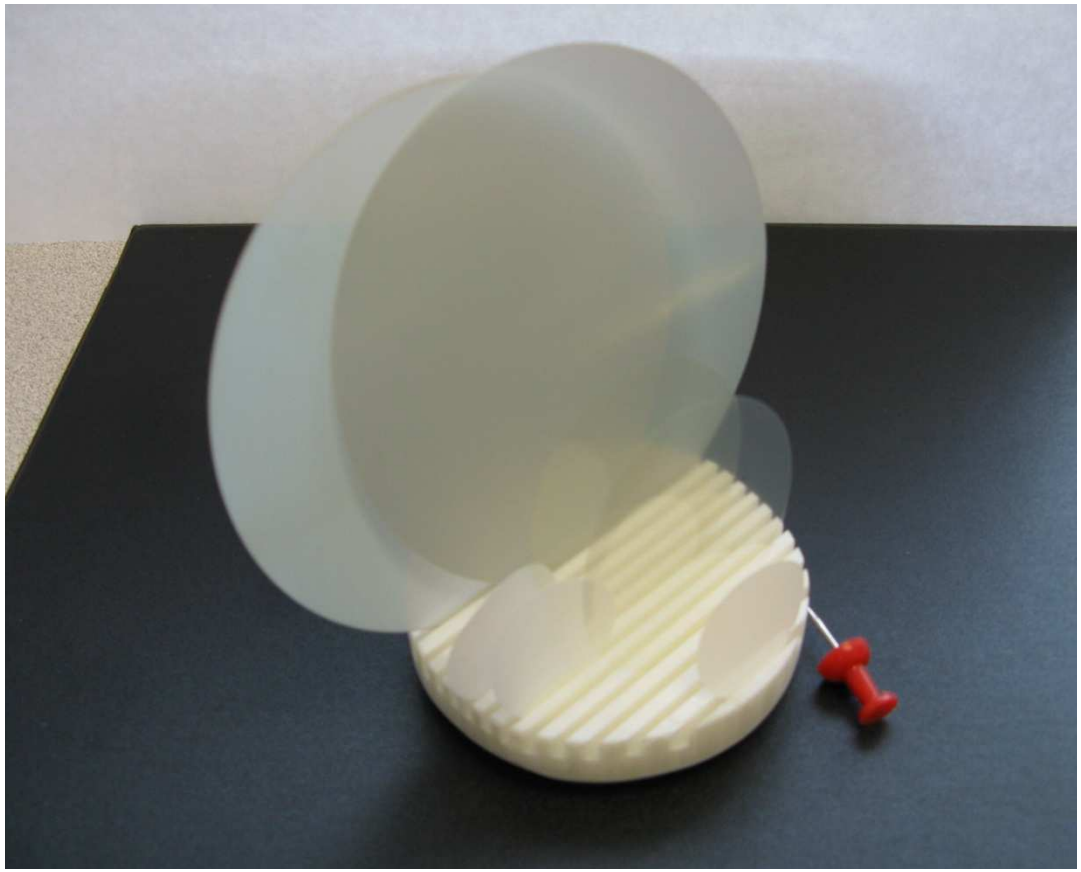
Assumptions for Initial estimate:

- Labor cost for high voltage processing is comparable to current processing
- Volume <10 m^2 /yr: current processing cost for 2.5”x2.5” Al for AAO-based membranes and sensors produced and sold by Synkera
- Scaling from 2.5”x2.5” to 8”x8” will to reduce cost per unit area (less handling)
- Volume >100m^2/yr: based on recent scale-up analysis for production of 1M and 10M units/yr of small (~1 cm^2) AAO-based membranes and sensors

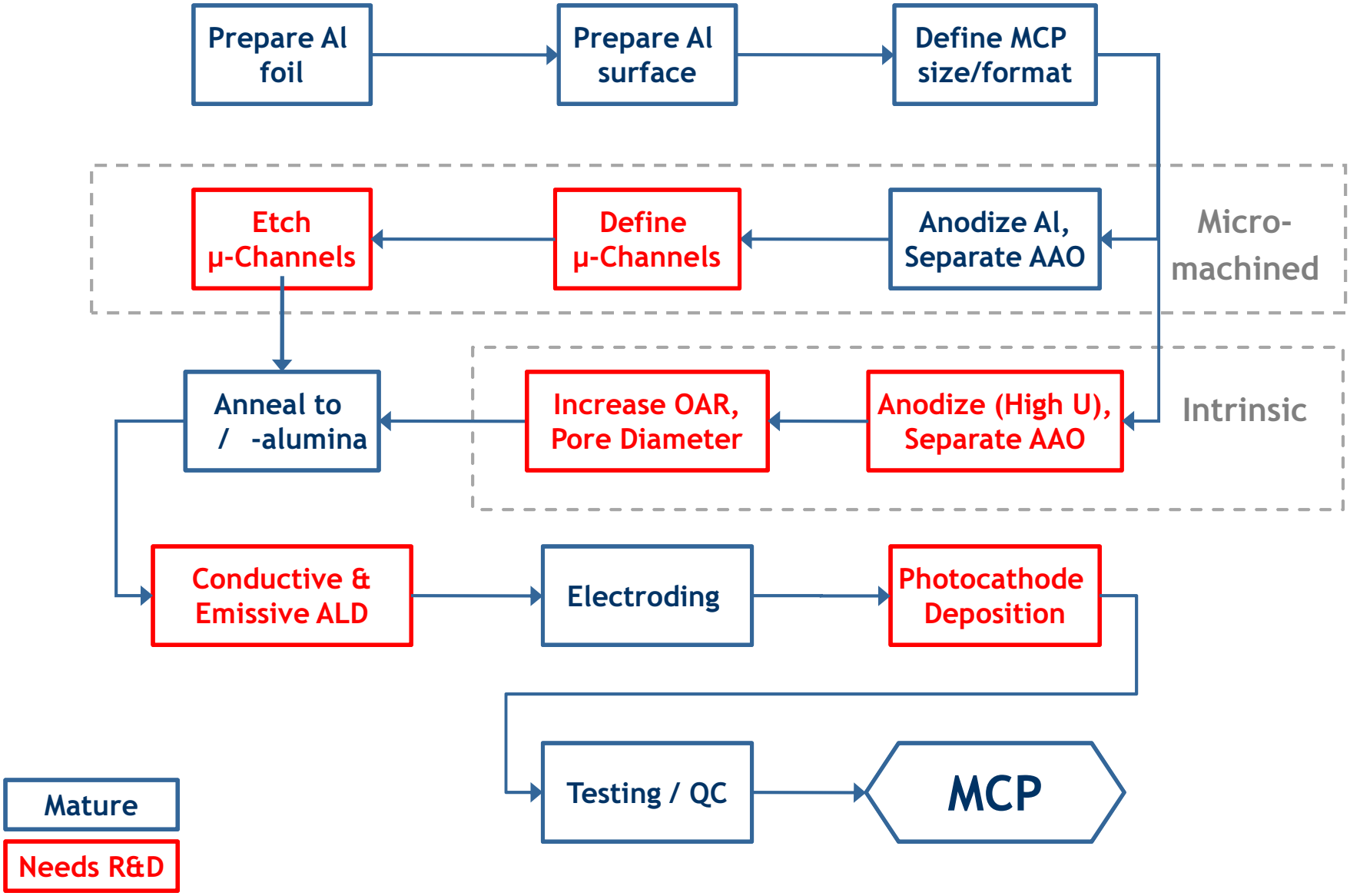
Projections:

Total Area. m^2/yr	**<1 m^2**	**10 m^2**	**100 m^2**	**1000 m^2**
Total 8”x8” units/yr	**<24**	**240**	**2,400**	**24,000**
• Price, cm^2:	<$10	$4-$5	$1-$2	<$0.5
• Est. Price for 8”x8” AAO:	<$4K (current lab scale)	$1.6K-$2K (current pilot scale)	$400-$700	<$200

AAP product example: 25-150 mm membranes



Readiness of AAO MCP Manufacturing



Remaining Priorities

Remaining for Year 1

- Fabricate and deliver new Batch 3 substrates to LAPPD team
- Target:
  - larger channel diameter
  - improved channel uniformity and alignment (moving into Year 2 objectives)
  - Functional MCP – gain demonsrtation

Year 2 – Functional MCPs

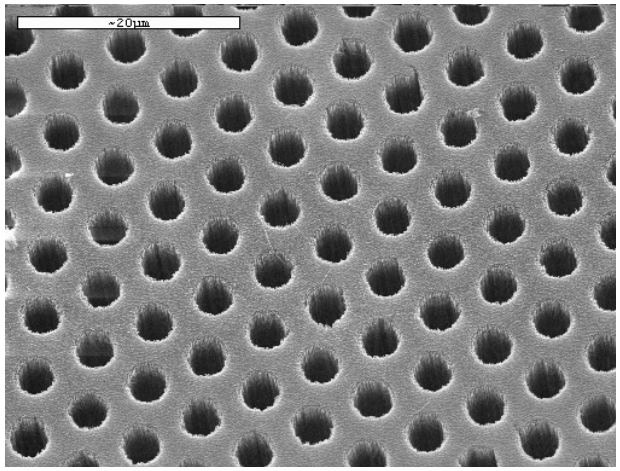
- Demonstrate channel diameter ≥0.7 μm and OAR≥65%
  (funnel opening and L/D 50-100 - default attributes)
- MCP substrates for LAPPD team (32.8 mm, qty ≥ 40) targeting above specs
- Optional: scaled 8”x8” “demo” substrates
- Validate cost projections for 8”x8” AAO substrates

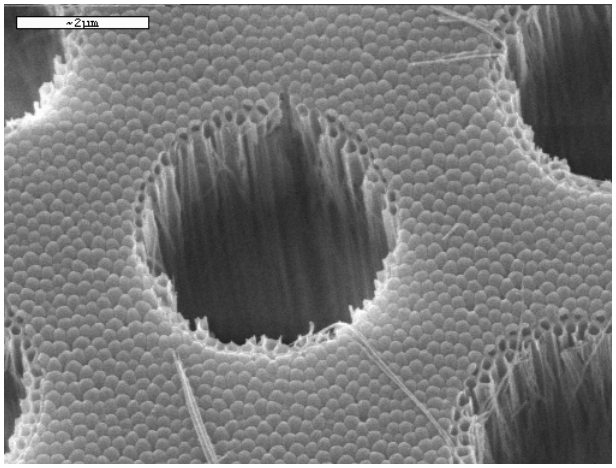
RELATED WORK

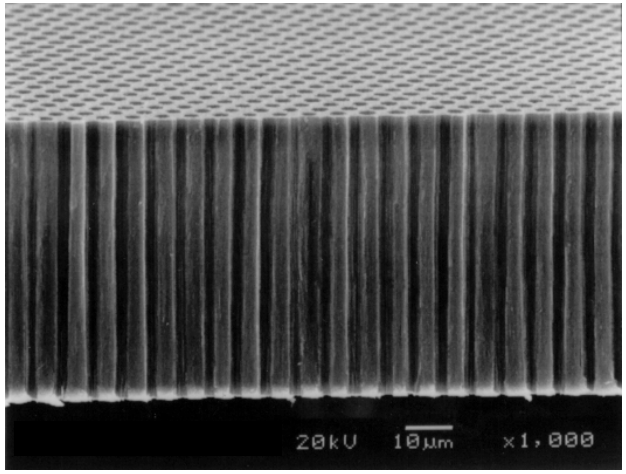
on

MICROMACHINED CHANNELS

HIGH RESOLUTION CHANNELS

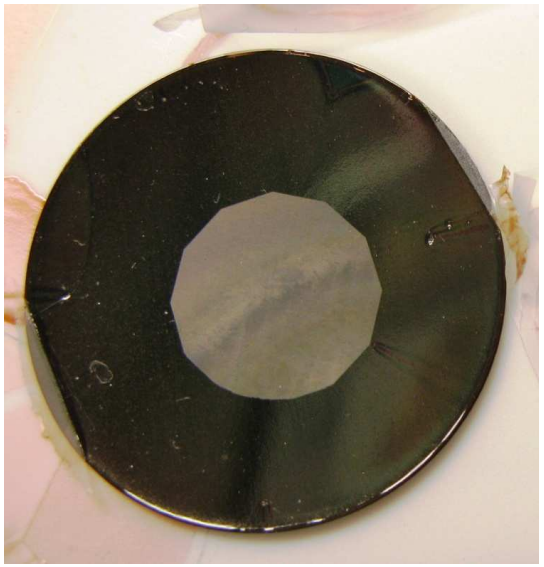






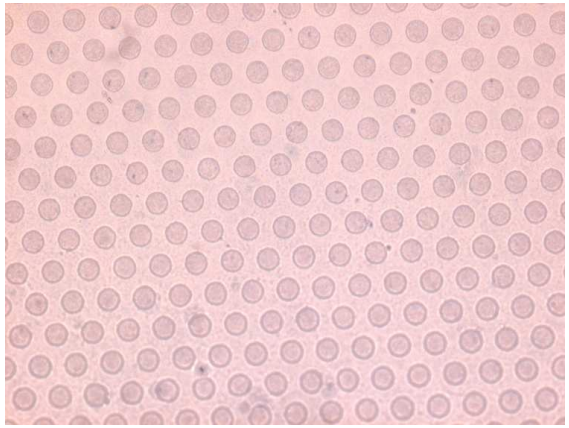
- Ceramic MCP substrate with ~3 µm channels, 60-100 µm thick
- Under development

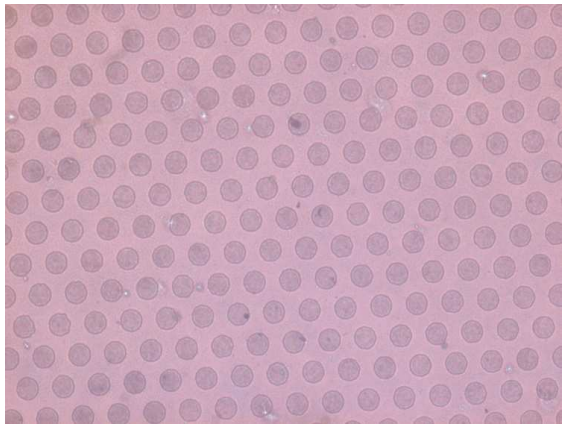
Prototypes with Micromachined Channels



After patterning

After etching





Optical image of 10 μm channels on both sides

Related work, not funded by ANL

- 25 and 32.8 mm substrates are being prepared
- Blank AAO is available for micromachining at ANL

AAO TO SUPPORT MICROMACHINING AT ANL

- Synkera can provide AAO substrates for micromachining at ANL
  - Conventional AAO, current product
  - Pore diameter: selectable (20 - 100 nm range, recommend 35-50 nm)
  - Format: 32.8 mm x 0.10 - 0.15 mm
  - Attached or separated from Al
  - With or without AL rim
  - If separated, barrier layer can be opened or closed

- Cost depends on volume and format

- Example:
  - Free-standing 32.8 mm x 0.1 mm “membrane” , barrier layer open

Quantities	5-25	26-100	>100
Updated Price, $/ea	$75	$50	$40

STATUS OF AAO-BASED MCPs

PARAMETER	CURRENT AAO MCP STATUS AT SYNKERA		RISK
	Micromachined	Native pores	
Max Size	25 mm prototypes in development	33 mm in dev't, 8" x 8" feasible	scaling new AAO processes
Development status	prototype development	prototype development	producing functional prototypes
Channel Size, µm	2-3 (for L/D=20) needs dev. for high L/D	up to 0.5 µm in 33 mm (potential up to 1 µm)	achieving high resolution in required size & AR
L / D	< 20-40 (for 10 µm channels)	10-200	achieving L/D with required resolution in practical format
Channel Bias	needs development	Feasible - YES, Practical?	validating in practical scale, is it needed?
Resistance	ALD processes proven feasible, needs development		fine-tuning the specs; reproducible in size
Temperature Range	up to 1100°C confirmed		none
Mechanical Durability	robust	very robust	scaling to size due to low thickness
Lifetime / Stability	unknown	unknown	validating assumptions
Competitive Posision	drop-in replacement	undefined (large area?)	lack of a thorough performance evaluation, validating assumptions

LAPD